Notebook, N-88-03-15.1

This notebook covers the period March 1888-April 1889. It was used by Erwin Von Wilmowsky for rough notes and calculations regarding chemical analyses and the preparation of chemical compounds. Included are analyses of chemicals ordered for the laboratory and assays of copper and nickel ores. The prepared compounds were designated for use in various experiments, including phonograph cylinders and insulation. The book also contains scattered entries relating to battery, filament, insulation, and chemical ore separation experiments. There is a table of contents at the beginning of the book. The spine is labeled "57." The book contains 186 numbered pages. Nine loose pages of correspondence, notes, and other material relating to Von Wilmowsky have been inserted into the book.

Only the material pertaining to battery, filament, insulation, and chemical ore separation experiments has been filmed.

1) Carbon Filaments from Ulmic Acid: dist 106 g dist + Carbon 193 g + H_3PO_4 233 g heated to 320: bubbles, cane got slowly, it is gel soluble in water, heated more. The substance blown up, is flexible & flexible also, easily, in soluble in water. Weight and Dec 12 loss 61% of 127% i.e. 58%.
The substance is transparent, very shiny.

2) dist 106,5 + Car 213,5 + Glycerine 253,5 + 209 Bar Ac cryst heated quick to 370° C. Threads made, soluble in water deliquescent.

3) dist + Car 179 g + Bar 186 g

Tungsten Electrodeposit

Tungstate Soda 3 oz + 16 oz aq
mixed by clean $K_2S_2O_8$ Solution
heated by water, then (1:5) Dil. HCl,
water again and redissolved in $K_2S_2O_8$ Sol
in excess of final) + 25% more.
Wolframic Acid + ¼ pt NaOH melted and
electrolysed with Carbon Electrodes.

White Deposit Ammonia
with Carbon Electrode
electrolysed from out
c 30° F Reduction of temperature

3 Filament samples: A Melasse Acid (Brown Ac) 41d + Carbolic Acid 5% heated 1 hour 190-160°C until gelatinized, loss 23% (Carbolic Ac and water). tough and flat.
B 110d Glycerin + 20d brown Acid heated in a double bottle until as evaporated
C Sugar + Carbolic Acid heated in open air.

2 Sugar + 2 Glycerin (distilled bp 290-360°) heated to 240°C until insoluble in CHH gelatinized on cooling with gas bubbles remaining.

Glycerin 4 lbs were distilled so that nothing could flow back in the still and a 70% nearly colourless distillate obtained at 290°-320°C 10% at 240-290° and the rest with temperature quick rising to 360°C very dark coloured and much decomposition going. The residue gave good filaments of more strength than the hydrocarbon but with much ashes too.

2 Sugar + 1 Distilled Glycerin heated only until insoluble in aq ether, a cylindrical condenser was used & good filaments if dried slowly and heated in dry CHH gas and carbonized in CHH ~~gas~~ too.

Allways (except one case) there remained gas bubbles in the mixture if gelatinizing took place and gelatinizing came even with much 2 Naphtol or Glycerin or both present (fr 1 pt to 3 pts Sugar remaining) and sometimes on cooling even.
Then the gas escaped even when cooled down so much as to 160°C.

The Sugar + Carbolic Acid + Glycerin ~~evaporated~~ partly and heated to 240° and then in Papin to 340°C and evaporated at common pressure from 190 to 24 pts gave a mass

quite free from bubbles and entirely
dangerous good for filaments.
but after carbonizing as well as
before the ~~carb~~ filaments were
more brittle as gelatinized
Sugar - glycerin.

1879 Filaments:

The best results filaments were
obtained from Sugar + glycerin + ClH.
Raw Sugar was omitted and the
Residues from making Chloroform
both by ClH as well as by Soda
were evaporated slowly to dryness:
these Substances gave good and
strong filaments without bubbles.
Glycerin simplified [illegible] in presence
of a Residue which was useful
Glycerin + 10% Silicon Dioxide
Lanolin Oxide, Lactic Acid Residue
Dioxide.
Industrial Lanolin saturated with SH_2 (A ab
Tarbs c 60 [illegible]) and evaporated.
Sugar + Glycerin heated (1 : ½) until insol
in water, also and during 3 weeks settled
down to a brown tarry syrup once more
body to the orange liquor contain-
ed residues and better filaments.
But Bamboo fiber dipped in gly-

cem and heated [illegible] Ac
exp the air [illegible]
with glycerin saturated with Cl
[illegible] pressure with the above
[illegible] dilt under [illegible]
[illegible] to be the best [illegible].

[illegible] to dell the big [illegible]
carbonator [illegible]:

Rosin 225 lb (Ca O) heated
Asphalt 4 lbs heated
Linseed Oil (strongers mixed with
the above equal parts gave [illegible]
[illegible] strengthening [illegible]
[illegible] of the mass of [illegible] 225°F.

NB. [illegible] Sheet
[illegible] Stearates take [illegible]
[illegible] dry Cl_2O_3 gas.

Pipe Filling Insulator:

1) Stearate of Lead from Stearic Acid heated with ½ Litharge made:
2) Stearate of Lime from Stearic Acid 100 g + Ca O C.P. 20 g heated, made.
3) Stearate of Lime (soluble in Turpentine) heated with Rosin Oil 2 min until combined to a soft gelatinous brittle mass
4) Stearin Pitch Jensen and Marshalls Stearin Residue from Morris or Motts Co fried.
NB. both carbonate well for filaments, the Pitch contains small amount of Ashes.
5) equal parts boiled Linseed Oil, Asphalt, Lime — Stearate gives good mass.
6) Lime Stearate will not ~~combine~~ mix with Linseed oil and the basic lime Stearate not even with Rosin Oil.
7) Rosin oil + 30% $Ca(OH)_2$ will not mix well because $Ca(OH)_2$ was sieved but rather too lumpy. to be tried the

paint mill for this purpose.
pag 72

China Cements

Several clay mixtures tried.

180 Kaolin
90 Feldspar
90 Silica
90 Na silicate Syrup

} stands well in fire, shrinks c 7% diam after wash c 9 tons pressure psi □".

Vitrite white, non porous, polished in ag 12 hours and tested stands sufficient resistance, does not flash more than air does.

As Na silicate was replaced by burned Gypsum the mass was porous. Stood well (Properties 88.190.26.12.8) Dicalcium silicate was produced by oyster shells.

Flux spar + Gypsum equal parts formed an easily fusible glaze.

Oyster shells tried. Fire Clay makes the mass more manageable, avoids fissures (50 25 12 12 + 4% ag) but requires more Feldspar.

tried 150 Kaolin 100 Feldspar
90 Silica 90 Na silicate Syrup
150 Kaolin 25 Feldspar 12 Silica
12 Oyster shells 12 Fire clay 8 ag

Filaments

180 g. Glycerin freed from Copper by SH_2 were heated 7 hours with c 380 g S_2Cl_2 dropping in at 130-140°C, then 3 hours 180°C boiled with a thorn condenser, cooled, filtered, evaporated left good Residue for filaments, stronger but not so fine as the Residue from distilling the Dichloridrin.

The latter residue ~~good~~ is better to heat bombax fibre in, with the purpose to fill the pores and give shiny surface.

Programme:

to test carbonized and non carbonized Bamboo filament and raw fibres under vacuum and then pressure in the Silicate paddes and the Glycerine sulphur residues not above c 240°C

Ditto with water + Trace Carbolic Acid

Ditto with the old Molasses Acid + Urea.

Bamboo fibres were heated under pressure in Glycerin: carbonized into tar not dissolved in Glycerin

2/in Carbolic Acid: all dissolved temperature was 1 hour c 900°C

Anthracite powder heated ditto with Phenol & Naphthal, Anthracen was not attacked.

China artists glazed with pure Feldspar burned the same time with the mass. The half dried cylinders dipped in the glaze mixture made with Gum Arabic. Result good, does not more than 1/4 so much as air float. Resistance of the whole cylinder was 50,000 to 500,000 ohm. Ground surface quite white and hardly porous at all. Edges partly melted, mass proved best if taken as 1 pag 56 (180 : 90 : 140 : 90).

Potassium Silicate Syrup Dido 300° C lost 60% aq and is then easily pulverisable.

Potato silicate crystals Dido lost 50% aq.

Kaolin was previously heated to redness for 2 hours and lost 1.8–15% aq, was powdered before heating. Good to reduce shrinkage.

Oyster shells lost on heating 2% formed Kalk with aq an not attackable paste, not much as a flux (an account of the fluoride lime in it).

Analysis of Lime for Bottlers.

SiO_2, Sugar, Fe sulphate were added to Baltimore Solution and on heating of the solution more than usual observed. Pencil marks gave the same result. Sh and As 10 mg to 20 g made the time boil in c 15 Min. Lime Fe nitrate with Lime was not tried.

Therefore Sh & As to be removed from the malt. Lime by a cheap preparation made as [illegible] Sulphur Solution of MgO_2 & Sugar are mixed with precipitated chalk salt dried slowly and gradually are mixed all water gases away [illegible] Hot. Equal weights of all the mixed, MgO_2 may be evaporated dry and gradually heated mixed only MgO_2 is left in fixed state and no more in compounds.

For Analysis of the Lime Under Marsh Apparatus is useless for quantitative work.

To be take 100 lb dissolved in 2 M H_2SO_4 + 5 times water + 5 % Cr_2O_3 (C.J. Calculated) according the formula $Zn + \frac{3}{2}Cr_2O_3 + 2H_2SO_4 = ZnSO_4 + Cr_2(SO_4)_3 + 2H_2O$
60 : 100 : 2.98

The "American" Chromic Acid contains 85 % CrO_3 we have to take '80 k instead of '67 pig 24

Chinaware Crucibles

Gypsum (burned) was tried as mud as c 20 % it made a solid mass only if a certain high temperature was not exceeded. At higher temperature the mass became quite porous.

With a red hot Rabbit and high pressure, full force of one man with long lever pipes on the big press (c 8 ton for plate of c 57 ") no shrinkage was observed, with the small hand press c 1½ %.

The Glaze was well when made 40 cc of 5 % gum Arabic 60 % Feldspar 6 % Phosphate of Lime Precipitated.

The mass was for a full yellow heat nearly too soft if made 150 Kaolin 100 Feldspar 90 Silica 90 Silicate Potash Syrup. (= 3 % 2/10 Dried 20 % C) and.

Bottle was 45 instead of 90 Silicate. No porosity in many more who then absorbed.

Burnt & precipitated Chalk to be tried. Magnesia etc. etc.

H

Analyses

Magnesium Chloride + Chloroform and
43.5 g dry + ($MgCl_2$ + 7H_2O) 1125 - 50 g
for remark, Ans as the Chlorine in other Lines: 618.2 g
Dish + Dish = 1542 g ∴ 3 oz $MgCl_2$ gotten
by heating about 4 ounces crucible until [illegible] had ceased
and no Chlorine vapours. Delivered to Mr Kenelly.

Chromic Acid produced (Courtesy Sydney Exposition)
from Theorspar, Lead Chromate, fig on strength
Sulphuric Acid (3 : 4 : 5) mixed in lead
Jar and the red gas (CrO_2Cl_2) + sufficient chlorine
to form $CrCl_3$ + HCl + H_2O. Gentle heat 1 : 1 : 3 fuming.

Chromic Acid Commercial Analysis
200 g German and American take 2 litres
spec wgts 1,126 and 1,130
made gold colour by wgts 55 cc and 65 cc
Ans aq. German precipitate Fe_2O_3.

$FeSO_4$ Sol made as ·507 N
15.7 cc = 7.95 N ≡ 3 g $CrCl_3$ Sol 20 g = 7.7 N Cr Sol
(therefore Fe Sol really only ·492 N. Average
taken in the following ·500 N.

German 20 cc used after dilution (1 : 10) 25
for 10 cc Fe Sol therefore ·247 N is the
German Cr Sol or 5 g Commercial CrO_3
contain ·247 = ·0619 eq g ≡ 2.06 Cr O_3
therefore 41.2 % Cr_2O_3 Standard
(≡ 28.8 % Oxygen)

Ames 2 N used 10 cc therefore 49 N
5 g ≡ 7.22 4.07 therefore 81 % CrO_3 (≡ 19.48 Oxygen)

300 g Cr SO_4 are contained in each cylindrical
barium cell. 1 Amphora ≡ 1.23 Cr O_3 Standard
300 × ·81 = 243 g ≡ 200 Amphora

Sulphuric Acid Standard S.G. 29 % SO_3
Free Acid for 1 % G. 140 cc Normal Ans 17 % SO_3
Ans 166 Normal g
therefore free SO_3 G. 24 % contained 5 %
Ans 16 %

Chromic Acid Heated gently and left Residues
German 66 % American 62.5 %
Salts hailed out for Residues (Potash) G. 11 % Ans ½ %.
N.B. ∴ 5 % contained SO_3 ≡ therefore only 11 % K_2SO_4
Arsenic found trace in Theorsar (4 %) and trace in German
(less than ·005 %) (very sh.)

Iron German $\frac{1}{10}$% American .015% Fe.
loss of 200° German 18,5%

German
~~Fe~~ O_3 .15
Cr_2O_3 41,2
Al_2O_3 11
~~Fe_2O_3~~ 24
CaO 29,3
MgO 1,35
MnO none
H_2O
Lead Chromate 100

American .02
81

$\frac{1}{2}$

$\cancel{17}$ 9,5 SO_3
$\cancel{1,5}$ 9,5 H_2SO_4
.003% As_2O_3, no H_2S
100

Pt Ore 22,640 } boiled with Aqua Regia
+ Au Cl_3 25,680 }
24,320

1326 $\frac{3}{4}$ 44,2% ~~loss Pt~~
61,5% ~~Pt~~ alone
5% loss of Pt by Ether vapours

Resin, Resin Oil I–III fractions, alone and with Stearic Acid were saponified by powdered CaO, ZnO, MgO and 10–20% Linseed Oil added.
This pulverpapaches were mixed c 1:3 with Asphalt, Coltarpitch, and Insulation, and c 10% Bees wax added
and the boiling point raised in this way.
But the masses were deemed to be less brittle below the freezing point.

Then Tannyl Acid (from Crude Palm oil) is to be substituted for Stearic. Sulphur is to be used and continued boiling.

Asphalt to be worked partly in Asphalt Varn (see Watt's Dictg) by heating 20–50 hours to 250° C.

Syrian Asphalt take $\frac{3}{2}$ $\frac{2}{2}$ $\frac{8}{140}$ Stick 9,8

Small Thermometer 9,2 hog 300° C = 1" from end
big " 9" from end
Heated 3 hours Max Summa 260° loss 8° = 5%

Temperature above = nearly 300° C.

Chromate Bath

Bichromate Soda 2 ℔ and Water 300 ounces 106
Con H_2SO_4 and additions of 21 % to equal Chromic Acid as this little contains 81 % CrO_3 and the formula 67 % Cr_2O_3:

Bath Solution 5 Chromic CrO_3 + 12,5 Con H_2SO_4 + 40 aq therefore to be replaced by

80 aq + 12 ℔ Bich Soda pure + (154+72 = 312 ; 17,2 = 66) Con H_2SO_4

try if this crystallizes

6 ℔ Bichr Soda 2 ℔ H_2SO_4 + 3 ℔ aq does not crystallize

100 lbs Chromic CrO_3 = ℔ 316 are equivalent to
121 ℔ Bichr Soda + 40 lbs Con H_2SO_4 add price ℔ 11,30
as the chromic crystals contain 17 % H_2SO_4 174 are to be added thus is ℔ 11,47.

480 − 64 = 416 ℔ Bichr Soda Con + 555 cc Con H_2SO_4 (50 spec 1,244 = 35,6 %) + 2277 l aq = 1139

or practically 416 ℔ Bichr Soda + 1 l aq

of this solution take 25 cc and to 500 cc

27,2 cc ≡ 20,7 cc ½ N Fe : (5/8 N Cr 12,5 cc)
≡ 9,40 ½ N Fe therefore Fe Sal 49 N)

then 27,2 cc Cr ≡ 10,14 N Fe : Cr Sal 27,6 N

Spec gravity Analysis 1,235
N Sal ≡ CrO_3 10,2/2,100 = 12,42 % l CrO_3
or 1415 × 20 = 283 % Sulphuric
283 : 416 = 68 % Sulphuric Con Bichr Soda
$Cr_2O_7Na_2 + 2H_2O$ contains theoretically 67,1 %
Sulphuric Acid only trace

Proportions are therefore

6 lbs Bichromate Soda	60 ℔	
15 lbs H_2SO_4	15	
40 aq	75	

equivalent to

5 lbs Chromic Acid	80 ℔
12½ lbs Sulphuric Acid	12 ℔
40 lbs water	92

75 % : 92 % = 81 : 100

If the above solution (500 cc 996 cc were left after filling Bath to mark, therefore 2564 cc were used, containing 75 % = 250 % or 81 % Chromic Acid

and 25,2 % = 645 g = 1,42 lbs 65 % H_2SO_4

2,2 oz Acid. Sol. 3,5 ¢ carbon 170 g = 1,94 AH
1,42 lbs Sulph. Ac 1,5 ¢ = 138 AH

Battery Zinc (½ plate = ½ lb = 6 ¢)
à 1,2 ohm Amp. Hours = 190 AH
meaning and 70 % of water
i.e. 0,4 ¢ for water
Amperage 4 ¢
5 ¢

Since 12 ¢ for 110 Amp. hours of Zn and H_2O only 20 % are left

Accordingly the figure Zn or H_2O be left
11,5 ¢ for 143 Ampere hours used 1,4 volt
19 % Wastage = 3,75 ¢ Zn & H_2O
Therefore 1 HP = 43 ¢ is all to be consumed
or 54 ¢ if only 80 % of Zn, H_2O and voltage is used

N.B. Ah consumed of the Jan 2564 °C of 1,240 sp. gr.
is 3180 g ~~carbon~~
11 oz Bichr = 6,9 ¢ = 172 Amp. H.
1,3 lb Acid. 1,8 ¢
½ plate Zn at 6. ¢
147 ¢

one Jan measured direct converted to Hanack 2420 °C
= 2420 . 1,1359 = 2,75 quart ¢ = 3000 g = 6,6 lbs
Bichr: H_2SO_4 : aq as 6 : 15,5 : 40

as 9,75 % : 25,25 % : 65 %
293 g + 756 g + 1951 g (since 3000 g)
= 10,3 oz + 26,1 oz + 68,9 oz (105,3 oz)
6,4 ¢ + 1,6 ¢ ≡ 199 g Zn H_2O = 162 AH
Zinc plate = ½ lb = 6 ¢
Since once filled 8 + 6 = 14 ¢ for 162 Amp. hours. Really it lasts about 20 hours = 45 Amp. hours
Remedy of the Zinc plates:
1) Mg in Acid; Mg H_2 (and casting with precipitated CO_2)
2) Sulphuric Acid free of Arsenic or Antimony to be used as from Poles a Nitrogen.
Consumed Battery Solution to be determined
The rest of Cu H_2:
This can be reduced by operating the motor so that a lower voltage will do, and increasing the diameter of the rotor.
Battery Leads 2,0 to 1,8 mm to the end (without increasing) (leading and returning as regulating lines)
Motor at rest ½ ω, while running ⅔ ω
Battery has resistance of 0,4 ohm. 0,3 ohm minimum, then in straight line increasing after 25 hours the end of life after 30 hours 0,2 ohm 15 ω and at the last is 0 after 34 hours 25 ω
Motor: (a) Battery ½ volt
(b) Battery ½ volt
months = ⅔ short lost 20 to 1,7 volt means 20 % decrease of power and 25 % increase 10 % decrease

To try: $HNaSO_4 + Na_2Cr_2O_7$

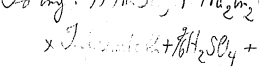
x Isomalt + 7% H_2SO_4 + 6% Bichromate Soda (heated to ...)

Electrical figures from last page show Alch. Chalk 25 lamps as standard time point as the proportion does and any more ~~[illegible]~~ will show A off (this comment) with 75 w Bottle-u and .33 Motor-resistance and 1,8 volt Battery – and .44 Motor-Electric Furnace. All only will work is 3,22 Amperes

multiplied by .44 volt = 1,64 watt

the Lamps map dar 2 49 watt

the Motor Battery and the ...



The original amount of Amperes is

N.B. Tar and Carbon gave 2 ... Exchange Jar. At first the preceding used less ... But stop for cheaper passed Carbon plates. – Bichromate Soda as above gives 1% Sur. Electrolyte place and about (½%) pure Hydrate and last 2% Sur. time.

The ~~[illegible]~~ of the scaling Motor resistance at full running at constant speed was as a average .66 w. ... end of ... is a account of variation ...

The cylinder ... 4¾" × 2" × ⅜" = 20 □"

4 Carbons ... 5" dipping in 1½" Depth ... 2 rows 1⅛" distant. Platinum – Carbon distance at the beginning ⅜ at the end ⅜ Surface 72 □"

At the ... the is 2,75 Amperes, after 25 lamp 2,3 ... 55 and 4 Dr. watt.

Try Amylsulfuric Acid in other I. Epharacids (not too high priced as Amylsulfuric is gallon (1 ℔) suggested by H_2SO_4 acids not ... on Amylic ...

Dichr. 535 g / 1225 | 10% Bichromate Soda of the same temp
" Bichr. 1258 | lost in the same time 13%
(1) 1213
(2) 45% = 6%
then 47 = 11%

Infusorial Earth Slake 679 g
Dried 654
loss 25 = 4%

260 g dry Bichr. + 756 g H_2SO_4 1,845 S.G.
+ 500 g Infusorial Earth dried and sieved

g become hot, smell of SO_2

27 g H_2SO_4 required more 12 g sieved not dried Infusorial Earth to give a sufficient dry paste

After preparation of Bichr. Soda 6 lbs + 3 lbs H_2SO_4 mixed by pounding and dried and thereafter separately 15 lbs H_2SO_4 + 7 lbs dried Infusorial Earth mixed and both put separately in air-tight bottles which must be dark tot on Infusorial Earth is heated in oxidising manner before.

Second Sample of Bismuth (Scotch)

[illegible] analysis [illegible] 12 [illegible]
[illegible] 2–2½ [illegible]
[illegible] 2½ [illegible]

The [illegible] of original [illegible] 55 [illegible]
[illegible]

819 g sieved Infusorial Earth heated
369 g heated in oven one hour
350 heated in oven two 5 %

(293 g Bismuth I (Philadelphia)
or 321 g Bismuth II (Scotch)) Bismuth dissolved in HNO3 and precipitated as basic nitrate
+ 200 g H_2SO_4

606 g H_2SO_4
606 g Infusorial Earth
(Both) 2 days (189 g flows 5°)
Will fill ⅓ of the space = 1900 cc

16 B. 6:15 p: 40 (see p. 84) from 19 hours only, instead 26 hours as 28 g with Infusorial earth only 17 hours. Water in left tube 6:45: 40 – 28 g Infusorial Earth.

Bath for 1951 cc aq (see page 85) 1 liter cell in

Chromate 3 g, Na_2CO_3 9 g + KNO_3 2 g.
Then 17 Cubic centimeters solution 1 hour
[illegible] gained 206 cc Chromate Solution, equal in color to 180 cc of 1 cc K_2CrO_4 Solution, which contains [illegible] Chromate Solution in [illegible] 1 % consumed. 14 Chromate Solution in [illegible]
Preliminary: Treated the same way once more gave 190 cc Solution = times 24 % of 1 cc K_2CrO_4 Solution
Summary: * 100 g ore will give 192 g Bismuth Soda crystal.

Theoretically is Cr_2O_3, FeO not = 224 4 Na NO_3 = 340
$2K_2CrO_4$ (= 3 [illegible]) : $Na_2Cr_2O_7$ + $2H_2O$ (= 29 [illegible]) = 100 : 26,7
Cr_2O_3 = 152 Na_2CrO_4 = 106

Then heat 224 g ore finely ground and sifted + same mixture of 106 g dry Soda + 300 g KNO_3 in porcelain crucible with oxidizing flame, stirring in water, add 3 Hydrochloric fuming, evaporate, crystallize and dry and wash in centrifugal.
164 kilos of 55 % = 55 % after 3 treatments and 309 CrO_3 found in Solution, this gives 1,68 (Bismuth Soda crystal) according to Abraham (the above only calculation text).
309 g CrO_3 = 309 Cr_2O_3 1,64 + 309 = 2,49 not all the chromate can be left in the filters. (3) Impossible a new plan. Analysis of real Chromate average.
Mistakes lost. Phenolphthalein.

For 9,75 lbs Cac are 3,6 lbs Tailings
i.e. for 1 lbs Bism are 2½ lbs and ore = 2½ necessary and ½ lbs = 14 Carbonate Soda dry and 14 Chlorhydric H_2SO_4

3/9 Chemical Earth sieved coarse 455 g + 10 cc H_2SO_4 [illegible] and heated in porcelain crucible over [illegible]

for ½ hour. Residue 323 g red colour [illegible] mass light. Added 246 g 66% H_2SO_4 C.P.

Dis 2535 } Bichromate 778 g Solid
" + Bichr 1313 } 742
Chloride } 742
Bricks 1277 } 1259 } 54

Taken 273 g (= 223 g [illegible] (page 86)) of this tail. Dissolved

Bichrom. + 78 g H_2SO_4 poured in and boiled once

more = half plate 55 g con. Lose + 3.4 g = 5.5 g

300 Liter + 190 H_2SO_4 con. lose 55 g = 14 g

986 − 771 = 1269 − 283 = 986 therefore loss of Bichromate 771 − 771 = 2 g instead of 12 g as $Cr_2O_7Na_2 + 2H_2O$ would lose.

[illegible] 223 [illegible] 10 cc 66% H_2SO_4 Commercial (1.84)

for 1 Bichromate 800 g H_2SO_4 instead of 801

3/12 120 g raw Chemical Earth sieved coarsely

30 g H_2SO_4 Commercial 1,848 spec

only a little SO_2, not much heated by mixing

continued page 101

Kaolin: Gypsum, anhydrous, hydrated, floated talc

absorb almost their own weight of H_2SO_4 while raw

Chemical Earth sieved absorbs 5 : 2, heated to 200° (300° long) absorbs 4 : 1 (good to heat it after weighing to [illegible])

For Copper determination 10 g $CuSO_4 + 5H_2O$ Sol

spec 1,065 10 cc = 10,65 g + the dilution to 106,5 g

in 1 cc 100 g Cu liquid — 10,5 10 g Sol. − 1,05 Cu with = 105.252 = 265 g

dilution will then amount to 265 cc 1 cc Cu in 1 cc

Dissolved (1889 1) 5 g 26,5 3,9 g Cu Bichromate Tailings No 1

(Raw 2) 12,5 − 19,5 = 2,35 Cu in 1 g [illegible] " Cone "

08 3) 12,5 − 6,8 = 5,7 g " 6 g Bichromate supernatant Cone No 2

34 4) same as solid 29,5 − 29,5 g Cu 3,0 g " Tailings "

12 g 8) " 144 144 g Experimental Bichromate No 2

9 g 9) " 150 g = 16,0 g Cu " " " 1

9 g 10) " 89 g = 9,9 g Cu " " 3

For Nickel determination was Fe as $Fe_2(OH)_6$ precipitated by Ammonia and then 1 g ammonia Nickel solution and Sodium Sulphide solution added found weighs 1,3 three − 1,8 134 − 12 9 9 g Ni.

Dissolved Bichromate mash 8.

The Earth Solutions for 500 g are amounted to weighs

for Nickel 1-4 each 50 cc for Nickel 8 & 10 112 112 152 cc

Organic 9 spec for Cu Ni Analysis 3 samples

for Mr. Dickson. Each taken 2 g floated Dis-

solved in dilute Nitric acid, precipitated by H_2S

filtrate 3 times precipitated by Ammonia and so all organic gone

Solution alone weighs for Nickel 25 1100 1100 1100 g

Ni determined by colorimetric with Sodium Ammonia

weighs 1,8 1,8 2,5 g Ni. Cu in colorimetric [illegible]

2,8 0,3 8,4 g Cu

Barkeiers (see pag 90)

2/12 300–400 Kollerei charges (same) ordered today
for each 300# fill 66° H_2SO_4 and 200# Infusorial
Earth. The kettle is heated to c 200°C & Lean
Alen mixed with the Acid and heated once more
as high as that for c 15–20 min in the air oven.
to be ordered 200 lbs Infusorial Earth
4 charges full strength H_2SO_4 for refiners
Geiler. Kettle empty 1399#

2/13 1 pail coarse Chromate ore contains (see pag 89)
19 % Cr (≡ 36,3 % CrO_3 ≡ 56 % Bichr. Soda equiv)
or 49,7 % Cr_2O_3. FeO
it was heated with ¼ pail CrO_3 freed & dried in
the furnace with Bunsen blast for c 6 hours
yellow heat for 3 hours. Mass was quite dark
while at the beginning white with only many
black particles. By H_2SO_4 or Sod. Solution the
CrO_3 was extracted. The overall mass was
entirely porous. The ore if ignited and not put
out but in cold water can be handpicked easily be pulverized. Coat the ore with Lime water and dry quick
on with NaOH into or mix it with Magnesium chloride
or Magnesium chloride (= $MgCl_2 + O$). Dry Barry &
Water Ford at the top of Decomposing Bath (c 650°C)

3/20 Chrome ore with Carb. Soda & CO_2 Ca precip. gave little result than with $BaOH_2$ aq instead of Soda. (76:62) but only here only 14% Bi-chromate (for 100 ore) instead of 56% by one treatment.
MnO_2 in small quantity may increase the oxidising or CO_2 in small quantity.

N.B. What is the usual price of Chrom. Ore one and Chrom. Lead one? (Cr in 44% or 30 to 40 per ton)

Fused 50g Chrome ore (sieve 80) + 3g Lampblack heated in Pt crucible.

N.B. Chromate + Thiosulphate Soda or H_2SO_4 heated to the result g mass after powdering $CaCl_2$ added and heated again.

Take 217g Ore (sieve 80) + 65g Cryolite well ground together and fused in 3 alternate layers with 80g Mixture of 64# H_2SO_4 + 16 Infusorial earth and heated in lead crucible.

Test. Infusorial earth + H_2SO_4 (2:5) 03 simply mixed stood 10 days
Then 30g 40 grs Bichr Soda (Bikarda) Sol + 33g conc H_2SO_4 + 62g aq = 125g Sum a
and 30g Bichr Sol + 46g Infusorial Acid + 62g aq 138g Sum
separate heated to 150°F = 64°C
5 ½ hour and both titrated 100:108
therefore 74% has by now leached the Earth.
On mixing 30g 40 grs Bichr Sol + 33g conc H_2SO_4 and cooling apparently nearly all Chromic Acid separated out, the mother liquor being only slightly colored. Cheap way to prepare Chromic Acid, by the mother liquor from a cooling can be freed from H_2SO_4 by crystallizing and the used again.

If the proportions of Bichrom H_2SO_4 aq are lbs: 16, 5 lbs: 4 Chrom Infusorial Earth heated 4, 1 reports leached 6.6 lbs will be there with no leached Earth a loss ~~of from the Earth~~ of 0.5 lbs = 4,5 + Bichr and and excess of Earth necessary of 2,5 lbs = 2,5 + Sum 7 + for in excess of the necessary 4, 1 lbs = 4,1 therefore heating is indispensable.

3/21 The above showed to Mr E. Baldenweck with regard, the best ale

cadmium 9 may 1897

Proposed $PbMnO_4$ and ask received to prepare it.

Literature Wagner '87 Regenerate by dark red heat, nal digester.

$KMnO_4$ boiled $= K_2MnO_4 + \frac{1}{2}O$

or in clay crucible or iron (see Zeyfferset)

5 g KOH 3,5 PbO_2 (or nitr. NO_3K) powdered + 4 g fine MnO_2 + 10 cc aq quick dried over lime. Lumps and some unattacked lead formed insol. soluble in aq.

Let cool a bit and keep on add water (not too much).

$KMnO_4$ + KOH + Alcohol give K_2MnO_4 even cold.

Used 2,63 g/cc $KMnO_4$ Solution containing 0,164 mol/l and an Acet Lead Sol. containing 1,39 Normal KOH 4,3 Norm.

a) Mixture 65,3 cc Mn + 25 cc K hydrid + 77 cc Pb + 10 cc HNO_3 (2N) + 8 cc Alcohol boiled.

b) 1 l Mn + 25 cc 7N HNO_3 + 110 cc Pb + 30 cc/ov Alcohol cast filtered colorless and diluted to c. 2 liter.

c) Some 1 l Mn + much KOH (10 Norm 1,3 N NaOH) + a little Alcohol (green a quick clear) + $Pb(Ac)_2$ and then much Acetic Acid added to redissolve $Pb(OH)_2$.

Question: is $PbMnO_4$ insol in conc NaOH?
can it be formed by heating Pb_3O_4 and MnO_2?
can Empirical $Pb(OH)_2$ flakes be transformed in alkaline manganate Alcoh. solution?
Can $Pb(OH)_2$ flakes transformed by Permanganate be reduced to Manganate by Alcohol?

$PbMnO_4$ = 325 will loose only 16 = 5% Oxygen.
Bichrom Sode required 50, this 160
Since about 10 % pb lb, can be regenerated.

d) 1 l = 164 mol. Among conc. oxydic 4 % Alcohol to Aldehyde or 2 % to Acetic Acid if used in alcoholic solution = 120 cc Pb Acet

Try flask Litharge + Manganate Alcohol and crystalline Litharge too.

$Pb(OH)_2$ conserved to a plate and then treated in Manganate solution + caustic soda.

or in conc Sol of pure Manganate Soda produced from the air dry KMnO4 or gasoline gas or etc.

Manufacturing Bichromate

for 200 lbs Bichr Soda

There are tak 600 lbs ore containing 22% chromogen part of 13% Chrom, to be mixed the chrom part with 90 lbs Cryolite and 100 lbs Ac Sulph and heated then added 200 lbs of Caustic Lime and roasted.

Then treated with Soda Solution and the Solution evaporated,

The ore was heated in the last experiment at store only floated ore was used at first to 400°C

for one 100 g floated ore } 3 hours
Lasted 50% Cryolite, 13% Ac Sulph } 200 - 400°C
separate larger ... the same vessel, to heated for 3 hours

then mixed with 50 g Caustic Lime and roasted at moderate-lively redness 3 hours, boiled with conc Solution of 14 g Soda, settled, decanted & evaporated to crystallization Result 32 g Bichromate Soda

Prot Bichromate Manufacture

1,2 tons chromogen per ton 3 tons ore

200 lbs Cryolite $25.00 $54
400 700 Ac Sulphuric $
600 " Chromogen $ 5,00
800 Soda $ 18,00
1½ ton Lime $ 9,00

2000 lbs $ 86
1 lb 4,3 ¢ price 9¢

If 44% Chrom ore Ton $40 is used
only 13/44 = 3,4 = 1760 lbs = $ 35,20 is needed
+ 600 lbs Chrom ore = 3,00 + 800 Soda = 18,00 + 2000 Lime = 5,00 lbs
Sum $ 61,20 = 3,1 ¢

The above figures delivered 3/29 and Experimental Plant Estimate and Drawings promised to deliver next week.

4/1 The 44% Cr ore can be purchased for $5 per ton in ship to Niagara Falls on rail. Centrifugal rollers to $150 size for $12 per ton. It is to be roasted in oven ($1 per ton) or if 10 tons $2 per ton if 5 tons in 29 tons per day. Cooled in revolving axes ($40 lining) and then to be leached in revolving furnace are mixed with 2-3% Na_2CO_3. Price of 95% to 98% Na_2CO_3 2¼-2½¢

Sodium bichromate 55% 13 6¢ | Soda 1¼¢ (Pure 95%) bulk
Potassium chromate 60% 18 | bulk ore 50% Cr contains 97% Na_2CO_3 for 1.05¢
Chromic acid 65 1¢

Chromate bulk than bulk of soda. Simpler furnace be used. The roasting is to be done at still red heat but not blazing red heat. The chromic acid can be prepared indirectly by Sulphuric acid and be centrifuged and washed on. CrO_3 can be decomposed by SO_2 or H_2SO_3 etc. or chromic acid can be decomposed by the Sulphuric. CrO_3 is also used for reducing CO_2 down to ~~[illegible]~~ coal is with CrO_3.

The Monochromate is transformed into Bichromate by CrO_3 prepared from one Monochrom Sal by H_2SO_4 centrifuged.

Chromate Bichloride from pag 103 109

3/30 6: 16,5 : 40
20,6 lead Dissolved on 27,2 not lead done
$11\frac{1}{2}$ = 15% 90% Brich Sal
% 21 cc aq
both Samples heated 9-11½ 152°F 11½-1½ 124°F.

9/6 Since a 2½¢ tested and found
Coarse Fe As Sb no Lead but in Sumna less than 1/100 % 2.99 dissolved, less than 20 mg in Sumna.

Filaments cable for pass

3/12 Sample
Cable Tell evaporated to 95% + pour + add 2675°
other Leading 3 hours 262,3°
cable be drawn into filaments at 150°C
is not enough elastic for the lamp filed at 200°C.
Thing more Leaded 256 + 15 = 271,5° nail can
scratch the melten mass, bon milling point
The mass drawn into filaments and heated carbonizes very nicely, quite strong, not much brittle
then heated gradually and to a very high temperature. Not to be calcined with the light carbonel material.

C. Iron

Wanted a 3/29: Molybdenite of Fine equal parts
fusible before the Blowpipe, fine grained.

1) Ammonia Sulphate: Chem News 20 191
$FeSO_4 + Na_2SO_4$ calcined and washed in Pt Crucible
in H_2 stream followed by acid Permanganate, alcohol Ether & conc H_2SO_4)
at 350°C reduced, then heated in air and the spongy Cu-
pressed to ½ vol and L Calcined with Oxygen free
metal.
Methods of Fe ($FeSO_4$ + $MgSO_4$ + $MgCl_2$) contains to much
gas (p, CO_2 etc)
Fe reduced must be spongy & entirely soluble in Dilute Acids,
2) Wöhler: CO_{all} 1263 Cover Fe fused in Zn.
3) Schlechtendal Jahrb 55 255 Alloys of Fe heated below
melting p. of Zn in Hydrogen. Soluble Fe alloys much
than ordinary temp: the Carbonyl to redness gives Nitride
$9FeCl_2 + 4NH_3 = N_2Fe_4 + 8HCl + 2H_2 + N_2$
The Fe_2Cl_6 sublimed saturated with NH_3 etc

36 N.B. 6 No 36-47 Ulmic Acid
prepared made from Cane Sugar, Maple Syrup.

36 Ulmic Acid

37 Metapellic Acid Crude heated to about 400°F.

38 Ammonia Salt of Ulmic Acid

39 Same as 38 but heated longer.

40 Carbon from Metapellic Acid

41. Carbon and Metapellic Acid Crude

42 Phenyl Biborate

43 Ulmic Acid nearly exhausted by NH_3 and H_2O and made according to receipt on page 3 Ulmic Acid I.

44 Made the same as 43 but heated for 3 or 4 days on steam bath

45 Ulmic Acid made at a low temperature?

46 Ulmic Acid made according to II page 3 ~~last portion~~

47 Made as 39 but another lot. Metapellic Acid and NH_3.

48 Biborate of Beta Naphthol. 123

49 } 50 } Samples of Metapellic Acid & NH_3

51 Metapellic Acid dried on mercury and on glass. 51a the same heated more

52 Catechin dissolved in H_2O with or without NH_3 Ulmate of Ammonia.

53 Catechin dissolved in H_2O but heated more than 52

54 Samples of Ulmic Acid made from Maple Syrup.

55. Ulmic Acid made from Caramel and evaporated partly with NH_3

56 Ulmic Acid most soluble in NH_3 made from Recipe No 1 page 3

57 } Tannic Acid 20
Potassium Sulfate 40

58 Al. palmitate neutral or a little basic

59 Ditto + S_2C.

Month 89 to April 1.'89 } **Ore Extraction** { Nickel and Copper.

Principle

1) Matte + S + H_2Cl_2 matte and in molten state passed by before a magnet and the fluid part so separated. at a temperature over 400°C, the next time dito with above melting point at 150° so that it is magnetic.

2) Ore or Matte pulverized and roasted to be magnetic and at 400°–500°C separated by the magnet. The Non part cold separated again in Ni-Copper oxyd and Silica and the magnetic part of the last separation broke up by $H_2Cl_2 + H_2SO_4$ and separated cold ace mine in NiSO and CuO or NiS and CuS after treating with Sulphur instead of H_2Cl_2.

Done

3) Wet way. The Matte concentrated by roasting with Silica pulverized very fine and mixed with finely pulverized $Fe_2(NO_3)_6$ intimately and heated to 120–200°C for 3 hours and boiled with water and dil H_2SO_4.

Done good indications.

4) Speiss forming: The concentrated Matte + Arsenic Bisulphide melted together removing S as completely as possible and then roasted with Silica to remove most Iron. The Speiss and Slag then contains all Fe nearly as arsenide.

N.B. $FeAs + FeS$ melted $= 2FeS + As$

All Fe as S is below, nearly all Fe and all As is above with As.